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PROGRESS REPORT

DEVELOPMENT OF LOW TEMPERATURE
DIELECTRIC COATINGS FOR
ELECTRICAL CONDUCTORS

Fifteenth Quarterly Report

BY

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April 15, 1965

FIFTEENTH QUARTERLY REPORT April 15, 1965

DEVELOPMENT OF LOW TEMPERATURE DIELECTRIC COATINGS FOR ELECTRICAL CONDUCTORS

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A measurement error of 0.25% is the maximum expected

Fifteenth Quarterly Report April 15, 1965

DEVELOPMENT OF LOW TEMPERATURE COATINGS

for

ELECTRICAL CONDUCTORS

INTRODUCTION

This report discusses the development of connectors designed for use at cryogenic temperatures. In addition studies of the characteristics at cryogenic temperatures of RTV silicone rubbers in compression are included because of their importance in the proposed connector design which makes use of an elastomeric material held in slight compression against a rigid insulation to provide the moisture seal.

The results of successful studies directed to methods for removal of ML enamel and H-film are also included.

CONCLUSIONS

Cryogenic Connector Deisgn

The tests on the modified Pyle-National connector have been included during the last quarter. The results indicate that to assure reasonable resistance to the penetration of moisture, silicone oil must be applied to both back faces where the wires enter the connector and preferably the whole connector should be soaked in silicone oil before the compression fitting is applied around the protruding rubber of both back faces. The use of silicone oil is a nuisance and somewhat messy. Moreover, a complete seal of all the wires has not been achieved. A large part of the moisture problem probably can be attributed to the very small creepage distances between conductors at the interface between the male and female parts. Nevertheless, for many cryogenic applications the modified Pyle-National connector is probably quite adequate.

In contrast the cryogenic connector design described in this report makes very effective use of the compression between elastomeric and rigid parts and provides relatively long interfacial creepage distances so that silicone oil is not needed to obtain a moisture seal. Moreover, if a moisture leak does develop around one conductor, the leak does not contribute to low leakage resistance in the other wires as can happen in the Pyle-National design.

To obtain an effective seal the insulated cable must fit the entrance holes in the connector reasonably well (the same is true for the Pyle-National connector). Moreover, it is better to use oversize than undersize holes which may result in cracking. With oversize holes it is possible to obtain a seal by applying an RTV rubber in the extra space before the compression can is fitted. Such use of the RTV rubber is not recommended and is also a nuisance for field application but the rubber will take care of a situation which is likely to arise in practice.

Both size and weight can be decreased with the modular "building block" concept inherent in the proposed design. The "peg in board" approach will help in achieving size and weight advantage. However, additional design effort is needed to "commercialize" the product. Modular Electronics Inc. has indicated enthusiastic willingness to undertake the necessary program and to supply commercial quantities of cryogenic capacitors.

Contact Resistance

In this report the major variations in conductor resistance through the Pyle-National connector have been conclusively assigned to contact resistance. Since the male pins are so uniform (just one was oversize and did not contribute to high resistance) it must be assumed that variation in spring tension of the female connector is involved. This variability or perhaps lack of spring tension may well be more evident at low temperatures. While the values of contact resistance as measured are not high enough to be disturbing from the operational point of view, it may be concluded that a functionally significant contact problem might develop at cryogenic temperatures following exposure to vibration or to other detrimental environments.

Removal of ML and H-film

The use of the Microflame oxygen-butane torch constitutes a break-through in the problem of removing the polyimide insulations. Fortunately it is adaptable to field use. As with any insulation removal technique, care must be used in its use. However, the danger of melting of the copper is probably less great than the danger of a nick in the copper with mechanical means of insulation removal. Moreover, melting is immediately obvious while a nick can more easily escape detection.

The ability to remove ML enamel with relative ease makes the use of such coatings, particularly on stranded wire, much more practical. The ML insulation can be removed from even very small wires without melting the copper. Success is achieved only by using a very small flame. The flame from a large torch cannot be controlled and should not be used.

OBSERVATIONS AND SUMMARY OF TEST RESULTS

Cryogenic Connectors

As stated in an earlier report the construction of actual prototype connectors to Advanced Technology Laboratories' design was assigned to a manufacturer of specialized connectors. In this way practical experience and know-how in connector design and manufacture could be obtained. The prototypes have been made by Modular Electronics Inc. with the enthusiastic cooperation of its president, Wally A. Gammel. Modular Electronics has expressed a willingness to make production molds and to quote on quantity productions of the cryogenic connectors. It is recognized that minor modifications will be desirable in a production model. The prototypes were constructed with the objective of providing at minimum cost a model upon which a production design could be based.

The prototype connector for four wires is shown in Photos 1, 2, and 3 (a nickel coin is included for size comparison). In Photo #1 the pins are shown at the right center and the receptacles into which they fit at the left center. To the right and left of the pins, the molded polypropylene blocks, into which the pins and receptacles fit, can be seen. The silicone

rubber cover seals can be seen above and to each side of the polypropylene blocks. The parts of the steel case are located at the far sides. The first step of the assembly is shown in Photo 2. The completed assembly is shown in Figure 3.

Drawings prepared by Modular Electronics Inc. are attached to this report as Figures 1-7. A detailed drawing of the outer case has not been included since for a production model it would be designed somewhat differently and would be made of either aluminum or stainless steel. Assembly instructions as provided by Modular Electronics Inc. are given in Table I.

While the prototype has been designed to accept four wires it could be made also for any number of wires. After the original concept was developed and given to Modular Electronics Inc., it was recognized that the design could be simplified. It is proposed that the single piece construction shown in Figure 3 be replaced by round rod or tube (instead of square as shown) inserted through holes drilled in a flat polyphenylene oxide (PPO) plate. Other suitable materials such as SP polyimide could be substituted for the PPO. In a multiple tier construction space could be saved and the overall unit made more compact. Moreover, greater flexibility in arrangement could be obtained. The square holes in the end and center seals (Figures 4 and 5) could, of course, be replaced by round holes which should produce a stronger and more easily molded structure. Undoubtedly a standard modular unit would be devised but the details of production models are not considered to be a part of this program to develop the principles upon which cryogenic connector design should be based.

Both the prototype and the proposed construction provide about & inch of interfacial* creepage distance between conductor and shield. This relatively long creepage path distinguishes the subject connector from others such as the Pyle-National connector described in previous reports in which creepage distance is only 1/32 inch. On first observation it might appear that the proposed design is inherently large and heavy. Mr. E. J. McGowan, who has been principally involved in the proposed design, has made a comparative

^{*} An interface between silicone rubber and polyphenylene oxide under slight compression.

TABLE I

ASSEMBLY INSTRUCTIONS FOR PA10174 AND PA10175 CRYOGENIC CONNECTOR

- 1. Strip four #26, #28 or #30 AWG wires, exposing .125 of conductor (see Instruction #3).
- 2. Insert the four wires through top side of hood (B1437), connector cavity being oriented in the same direction as the stripped conductors after wires have been inserted.
- 3. Insert the four wires through end seal (B1436), connector cavity again being oriented in the same direction as the stripped conductors after wires have been inserted. (Wires may be stripped in this stage.)
- 4. Place contacts (A1432 or A1433) on stripped conductors. Crimp with crimping tool supplied by MODULAR. If soldering is introduced, extreme caution must be taken not to overheat solder joint presently located between crimp barrel and remainder of contact.
- 5. Insert individual contacts (connected to wires) into connector block (B1434). Caution: Insert pin or socket portion of contact into the end of the block that has the shallowest counterbored hole. The bottom of that hole has a slight chamfer to accommodate the chamfer on the large head of the crimp barrel portion of the contact. The opposite end of the block has a squared counterbored hole to accommodate the locking clip of the contact. After insertion, head of crimp barrel should be flush with top of connector block.
- 6. Strip end seal and hood down onto connector block.
- 7. Connector is now assembled.

analysis of weight and size as shown in Table II. If advantage were taken of the compactness possible in the round instead of square rod construction, it is probably that the proposed construction could be smaller and lighter than conventional connectors as exemplified by the Pyle-National construction.

The new commector has been evaluated by measuring both leakage resistance between pins and case during exposure cycles as follows:

- 1. Room temperature (23C and 50%RH).
- 2. Thermal shock by immersion in liquid nitrogen.
- Slow warm up to room temperature with frost first developing and then melting.
- 4. Thermal shock by re-immersion in liquid nitrogen.
- 5. Thermal shock by immersion in water at 23C.

Both extruded Teflon and unbonded H-film wrapped wire has been used. No problems are encountered with any of the steps above except immersion in liquid water. It was discovered that the holes in the silicone rubber seals were too large and a tight seal was not established. With water immersion the leakage resistance decreased rapidly to about 10 megohms (ionic contamination might have caused a further decrease). With melting frost a minimum leakage resistance of 2.5 x 10^{11} ohms was obtained which shows that water immersion is a much more searching test. Using extruded Teflon wire a clear PVC (Tygon) sleeve was slipped over the extrusion so the combination would not be loose in the silicone rubber seal. Unfortunately the combination of sleeve and wire insulation was now too large and the silicone rubber around one of the four leads in the rear seal cracked. Nevertheless, the cyclic tests were conducted. After water immersion for 15 minutes, the lead, about which the cracks had developed, showed a leakage resistance of 1.2×10^8 ohms while the other values were from 3×10^{10} to 3×10^{11} .

Another connector using a shielded H-film wrapped cable (#639-3, see Quarterly Report #12) was evaluated with the cyclic procedure. In this case the wire was also too small so that it fitted loosely in the silicone rubber seals. To overcome the problem, the leads were sealed into the rubber blocks with additional RTV-511 silicone rubber. In this case

TABLE II

COMPARISON OF NEW CRYOGENIC WITH PYLE-NATIONAL DESIGN

	Proposed Cryogenic Design (Prototype by Modular Electronics)	Conventional Design (Pyle-National)
Number of Contacts	7	55
With case - Weight Length Width Height Volume	40 grams 1 1/2 inches 1 3/16 inches + flange 3/8 inches	166 grams 3 inches 1 1/2 inch diameter + flange 1 1/2 inch diameter 5.3 cu. inches
Without case - Weight Length Width Height	13 grams 1 1/2 inches 1 1/16 inches 5/16 inches	-,-
	Calculated	
Number of Contacts	56	
Without case - Weight Length Width Height Volume	13 x 14 = 182 grams 1 1/2 inches 1 1/16 x 2 = 2 1/8 inches 5/16 x 7 = 2 3/16 inches 6.95 cu. inches	

the shielding braid was stripped back since a trial indicated that moisture would find its way along the unimpregnated braid as was expected. After 16 minutes immersion in water (after the other cycling steps) one value of leakage resistance dropped to 10 megohms but the others ranged from 6 x 10^{12} to 1.6 x 10^{14} ohms. Visual examination revealed that the H-film wrap in the one case had tended to unwrap just slightly but apparently enough to permit water penetration.

The test on the cryogenic connectors are being continued for longer periods of moisture immersion and a new specimen with Teflon wire sealed with RTV will also be evaluated.

The water immersion test on the modified Pyle-National connector described in the previous quarterly report has been continued and measurements made after 24 and 64 days immersion. The wires were measured in groups of 6 or 7 connected together. When a low value was discovered the individual wire with the low value was found and measured separately. The range of measured values of leakage resistance for the "good" wire groups is shown below:

Time of Immersion	Range of Leakage resistance - ohms
30 hours	5×10^{12} to 3×10^{13}
24 days	8×10^{11} to 5×10^{12}
64 days	5×10^{12} to 2×10^{13}

It is apparent that leakage resistance does not decrease significantly with increasing time of immersion. The "poor" wires all had values ranging from 10^6 to 10^8 ohms but curiously the same wires were not always "poor" as shown below.

Time of Immersion	Designation No. of the "poor" wire
30 hours	#37, 41, 44, 47, 49 and 55
24 days	#17, 37 and 39
64 days	#17, 37, 48 and 52

Why some of the wires "recovered" is unexplanable. It should be recognized that even the "poor" wires maintain relatively high values of leakage resistance which would not adversely affect the performance of much electronic equipment.

Contact Resistance

In the last quarterly report, variations were reported in the contact resistance in liquid nitrogen for the Pyle-National connector. In order to remove any influence of the connector itself, 24 pairs of leads containing the male and female fittings were assembled outside of a connector. It was quickly observed that the pressure to obtain a firm fit in the connection varied quite noticeably and a few of the connections were obviously loose even after the best fit had been made. The distribution of the resistance values for the leads at 23C is plotted in Figure 8. These results can be compared with Figure 3 of the last quarterly report (Page 14). A comparison is also shown below taken from the two charts.

	Resista	nce-Ohms at	23C	% Cha	nge
	10%	50% (PA)	90%	10%	90%
Wires (spearate)	.02443	.0247	.0252	1.1	2.0
Wires in connector	.02345	.02345	.0245	3.0	4.5

Apparently, as might be expected the variation in resistance is less in the connections separate from the connector where better contact could be assured. By comparing the probability plots it is apparent that skew is present in both results.

When the leads were immersed in liquid nitrogen it was discovered quickly that the measured value depended largely upon the amount of lead immersed in the liquid nitrogen. In consequence all of the reported values have been measured with the leads totally immersed. Unfortunately, this source of error had not been discovered when the measurements reported in the quarterly report were made so a comparison cannot be made. A comparison can be made with the room temperature values as given below.

	Resis	tance-Ohms		% Cha	ange
	10%	50%	90%	10%	90%
Wires (separate) at 23C	.02443	.0247	.0252	1.1	2.0
Wires separate at -196C	.00338	.00362	.0052	0.7	43.7

It becomes very clear from these results that contact resistance is the cause for the skew in the results so that about 30% of the measured values are higher than would be expected on the basis of the resistance in the wire and the soldered joints. At low temperatures the resistance of the wire is low so that the effect of contact resistance becomes very apparent. It is possible to make still further comparison by examining the values for a group of four wires with the highest resistance at 23C and a group with the highest resistance at -196C.

Resistance-Ohms

		at 23	<u>C</u>			at -1	L96C	
4 Wires with	<u>1</u>	2	3	4	1	2	3	<u>4</u>
Highest Values at 23C	.0251 (#15)	.0251 (#12)	.0255 (#7)	.0268 (#16)	.0037 (#15)	.0045 (#12)	.0037 (#7)	.0039 (#16)
Highest Values at -196C	.0251 (#12)	.0245 (#10)	.0246 (#13)	.02475 (#5)	.0045 (#12)	.0050 (#10)	.0055 (#13)	.0175 (#5)
For all wires								
50% value		.0	2475			.003	6	
70% value		.0	2491			.003	7	

It becomes apparent that of the four wires with the highest value at 23C, only one (#12) is also in the high group at -196C. However, all of the four wires with the highest values at 23C do lie at or about the 70% value in liquid nitrogen (-196C). In contrast, all but one of the wires with the highest values at -196C have values of resistance at 23C at or below the 50% value.

From these data three observations can be made:

- 1. High values of resistance at room temperatures can be attributed to high contact resistance.
- 2. High contact resistance detected at low temperatures may not be noticeable at room temperature since in some cases it can be "hidden" by the resistance of the conductor.
- 3. However, the contact resistance at low temperatures may be higher than it is at room temperature (i.e., wire #5).

By accident the connection in wire #5 was stepped on. In measurement the resistance in liquid nitrogen decreased from the original value of .0175 ohms to .0035 ohms. Three other connections with high resistance were then broken and remated several times in an effort to decrease the contact resistance with results described below:

Resistance at -196°C-Ohms

Specimen	#12	#10	#13	# 5
Original value	.0045	.0050	.0055	.0175
Value after "reworking"	.0038	.0035	.0048	.0035

These values after "reworking" are plotted with the designation X in Figure 9. Apparently only specimen #13 has retained a high value of contact resistance.

All but one of the male pins in the connections were found to have a diameter at 23C of $.0405 \pm .0002$ in. The exception measured .0412 in.

Compression Studies in Liquid Nitrogen

Since polyphenylene oxide and RTV silicone rubber were chosen as the materials to be used in the cryogenic connectors, it was recognized that some information about mechanical performance at cryogenic temperatures might be useful. With this objective in mind thermal expansion tests were made. The results are described in the last quarterly report. During the last quarter

semi-quantitative tests have been made on compression characteristics in liquid nitrogen. Most of the work has been limited to the RTV silicone rubbers after it was discovered that PPO did not crack even in 2 inch sections when immersed in liquid nitrogen. Moreover, at liquid nitrogen temperature PPO was not damaged by impact from hammer blows and in fact appeared to be almost indestructible. Silicone rubber and Teflon on the other hand in very thick sections sometimes did crack upon being immersed quickly in liquid notrogen. Silicone rubbers are also relatively easily shattered by hammer blows while in liquid nitrogen. Consequently, emphasis was placed on the likelihood of mechanical failure of silicone rubber which had been placed in compression at room temperature before being exposed to cryogenic temperatures. Quantitatively crude but very effective tests were made by compressing 1 inch long by 4 inch diameter pieces of RTV silicone rubber between the anvils of a C-clamp until the length was reduced to 0.55 inches. Greater compression would often result in fracture at room temperature. The compressed rubber in the C-clamp was then thrown into liquid nitrogen. Upon coming to liquid nitrogen temperature the rubber slipped out of the jaws of the C-clamp despite the very considerable compression at room temperature. The compressive strain "froze in" the rubber and additional thermal contraction more than accounted for the thermal contraction of the steel C-clamp. When the compressed rubber was removed from the liquid nitrogen, the outside surfaces expanded rapidly while the inside was still cold producing variations of the curious shapes sketched in cross-section below.





Despite repeated tests, fracture was never obtained in the silicone rubber during these severe tests. It is recognized that even more significant tests might be made by varying the geometry of the test specimen and by making specimens in which the rubber was cast around inserts of PPO or metal.

Attempts to make reasonably quantitative compression tests led to interesting results. A simple static loading device was built of Invar (low thermal expansion steel) which could be used with the test specimen immersed in liquid nitrogen and the movement followed by a dial gage actuated by an Invar rod. The device was very inexpensive and accurate enough for the intended purpose. Maximum error was in the order of .002 inches displacement when differential expansion was taken into account. Typical test results are described in Table III.

From Table III it is apparent that the room temperature compressive characteristics of the RTV silicone rubbers are quite similar but the ground silica glass loaded Adiprene rubber has lost much of its rubbery characteristics. The Teflon as expected does not display a true elastomeric characteristic. In the tests described at the top of Table III, the loads were applied as rapidly as possible, in about 15 seconds, until the maximum load was reached. The 53 psi stress was held and compression continued to take place but in large measure was completed in 10 minutes. This compression set is small and the differences between the RTV silicone rubbers is probably not significant.

It is interesting that the thermal shrinkage of the compressed specimens following immersion in liquid nitrogen is very much like that of the unstressed specimens measured in the dilatometer as described in the 14th quarterly report. These data from the test report are repeated at the bottom of Table III for comparison purposes. At nitrogen temperatures the load can be removed from the compressed specimens with so little recovery in length that it is within the error of the measuring equipment. Similar results were obtained when the load was reapplied. The latter results are not reported because the error hides any significant differences. It is apparent that even the silicone rubbers have a high elastic modulus at liquid nitrogen temperature and a high degree of strain developed at room temperature will remain "frozen in" at very low temperatures. It would be most interesting to determine the point at which the "frozen in" strains would release as temperature is increased. The rate of release would also prove to be very interesting.

TABLE III

DIMENSIONAL CHANGE*

COMPRESSION UNDER STATIC LOAD

% Compression at 23°C

Load PSI	RTV511	RTV11	RTV577		diprene % Silica	TFE Teflon
13	5.25	4.2	3.7	3.7	0	0.1
27	10.0	7.8	7.2	7.0	0	0.2
40	14.15	11.25	10.55	10.15	0.2	0.35
53	17.95	14.4	13.65	12.75	0.3	0.55
10 min. at 53	18.55	14.7	14.3	13.0	0.4	0.65
(Amount of set)	0.6	0.3	0.65	0.25	0.1	0.1
			e from 23°C t			
53	3.3	3.3	2.2	2.9	1.0	3.0
		% Recovery	in Length at	-196 ⁰ C wit	h Removal	of Load
0	0.1	0.15	0.25	0.25	0	0.2
		% Permanen	t Set after I	Return to 23	°C - No Lo	<u>ad</u>
0	0.1	0.1	0.25	0	0	0.25
			n from -196 ⁰ (rison - From			
0	2.8	3.1	2.4	3.0	1.2	2.2

^{*}A measurement error of 0.25% is the maximum expected.

Removal of ML and H-film Coatings

After many futile attempts a successful technique for removing ML enamel and H-film tapes from round wire and flat ribbon cable has been developed. This technique makes use of a small hand held torch (Microfilm, Inc., Minneapolis 24, Minn.) which produces a tiny butane-oxygen flame. The tiny, very hot flame is carefully applied so as to char the ML or H-film insulation without melting the copper. It is possible to avoid oxidizing the copper by applying the flame first to an area back from the cut end as shown in Figure 4. The charred H-film appears to prevent copper oxidation. In the photo the flame is being applied to FEP bonded H-film ribbon cable. With ribbon cable it is possible to pick up the edge of the charred H-film with the fingernail and strip it back. A thin film of FEP remains on the copper conductors and on the copper gage shield. However, the FEP need not be removed since it acts as a flux in tinning the copper either with a soldering iron or when dipped in a solder pot. With round wires, the whole length of insulation to be removed must be charred. It is important to avoid heating the cut end of the wire since oxidation of the copper will occur. Instead a small length of uncharred wire at the end may simply be cut off.

The charred residue of H-film on ML enamel on round wire is best removed with light sandblasting. A simple inexpensive spark plug cleaner is easily adapted for this purpose. However, the char may be removed with care by using abrasives and other mechanical means.

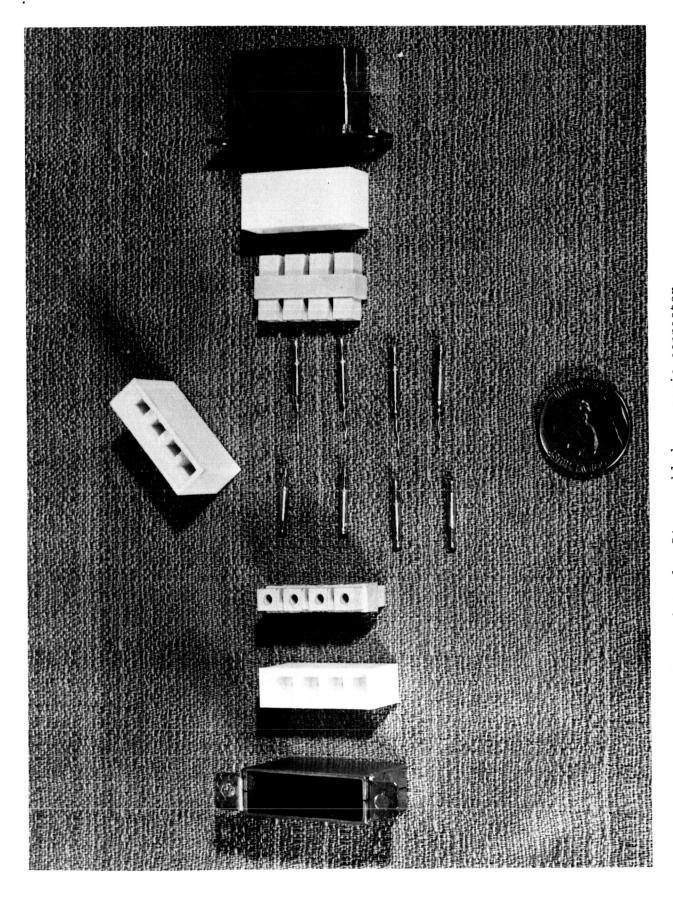
The small butane-oxygen torch is adaptable for field use but can of course be used in mechanized operations also.

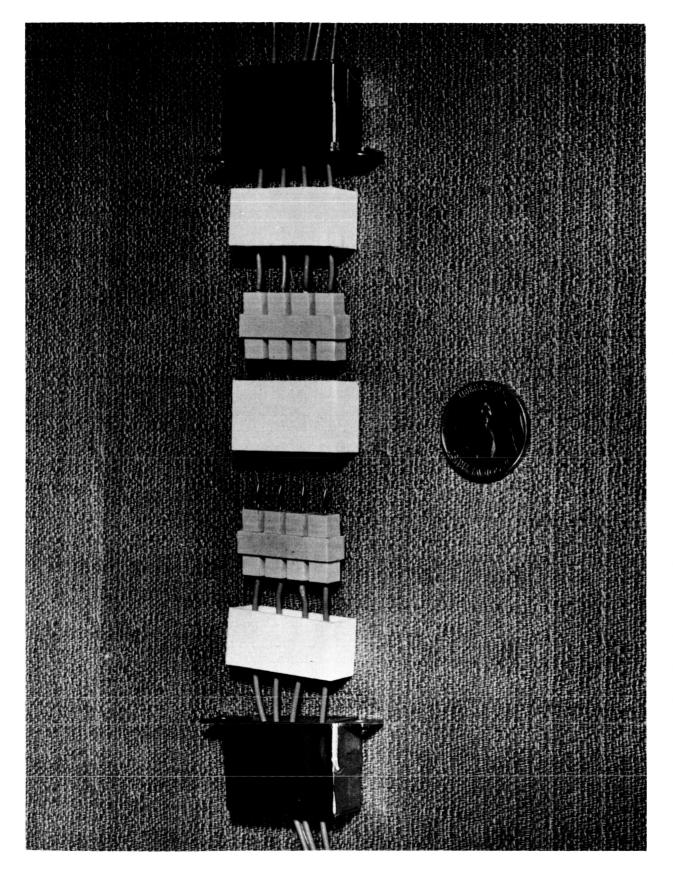
PROGRAM FOR THE MONTH OF APRIL AND THE SIXTEENTH QUARTER

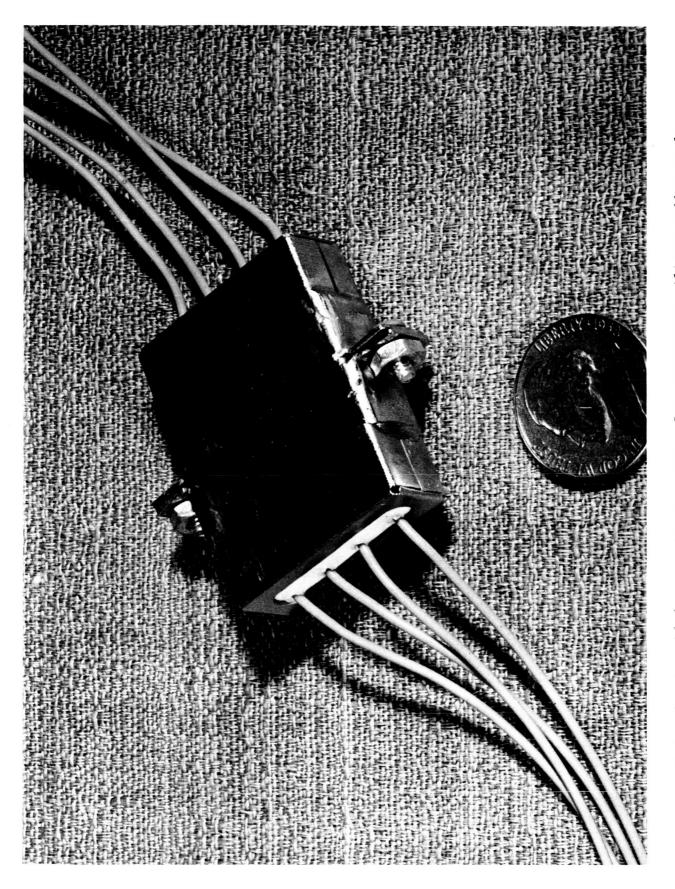
During April the design of a cryogenic connector for the flat ribbon cable will be completed. A prototype will be built in the laboratory and evaluated following a combination of cryogenic shock and water immersion. If time permits several prototypes will be obtained from Modular Electronics Inc. and evaluated for as long as possible.

The tests on the modified Pyle-National connector will be discontinued. However, additional cryogenic shock and moisture tests will be conducted with the connectors for round wires received from Modular Electronics. A laboratory prototype of a connector for round wire will be constructed using the rod and plate approach. It is hoped that SP polymer can be substituted for PPO in the new design to evaluate its suitability in combination with RTV silicone rubber. With the use of SP polyimide polymer the connector also becomes suitable for use at high temperatures. The maximum use temperature will be limited by the characteristics of the silicone rubber.

To the extent that time permits, additional compression studies at cryogenic temperature of silicone rubber molded about a PPO or SP insert will be studied.







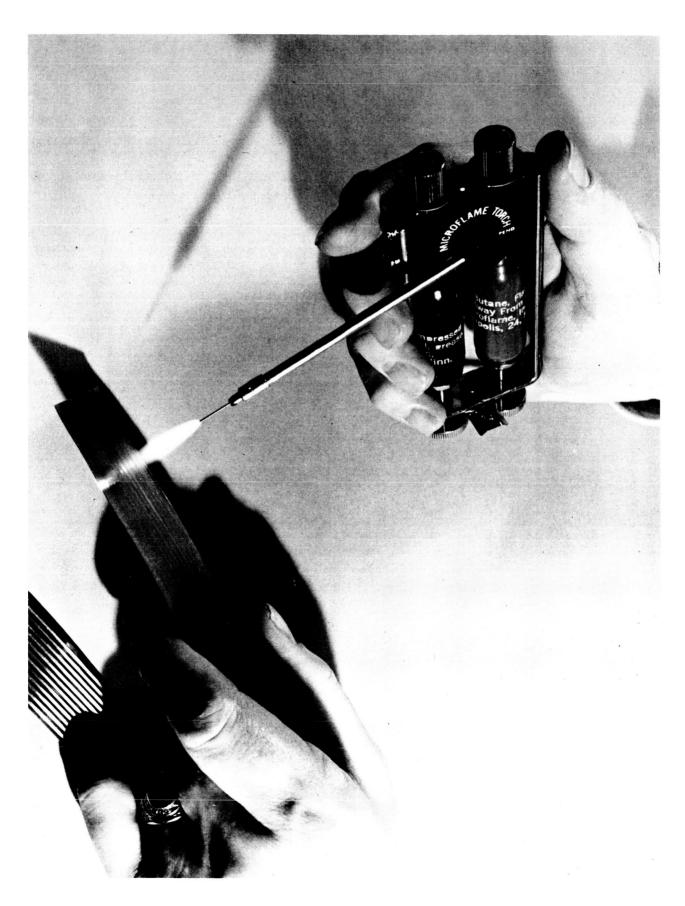
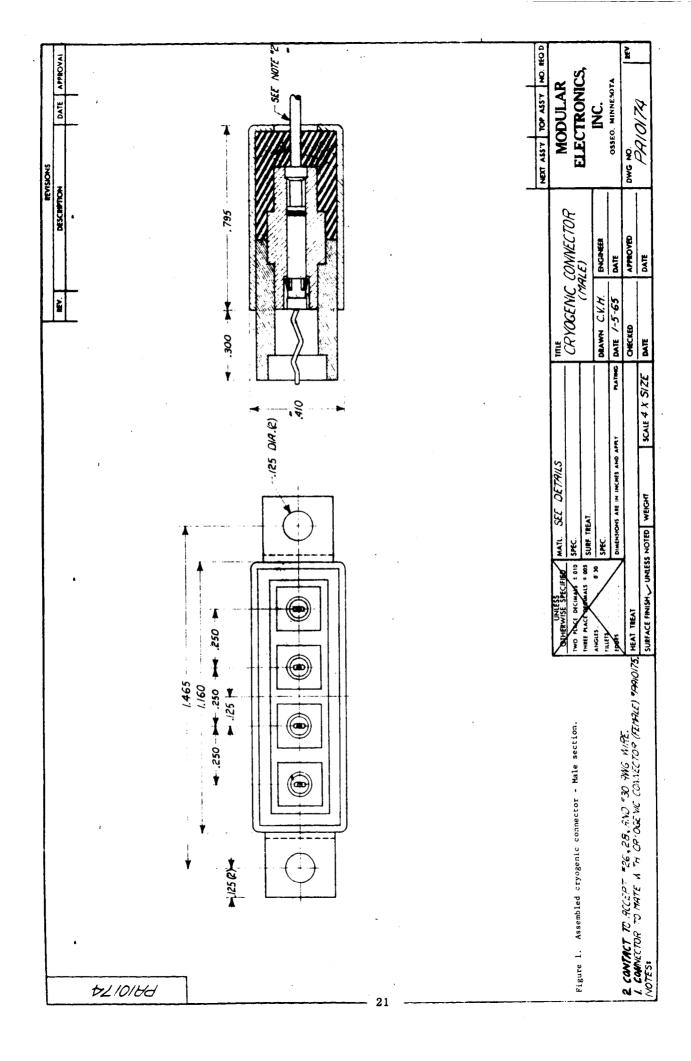
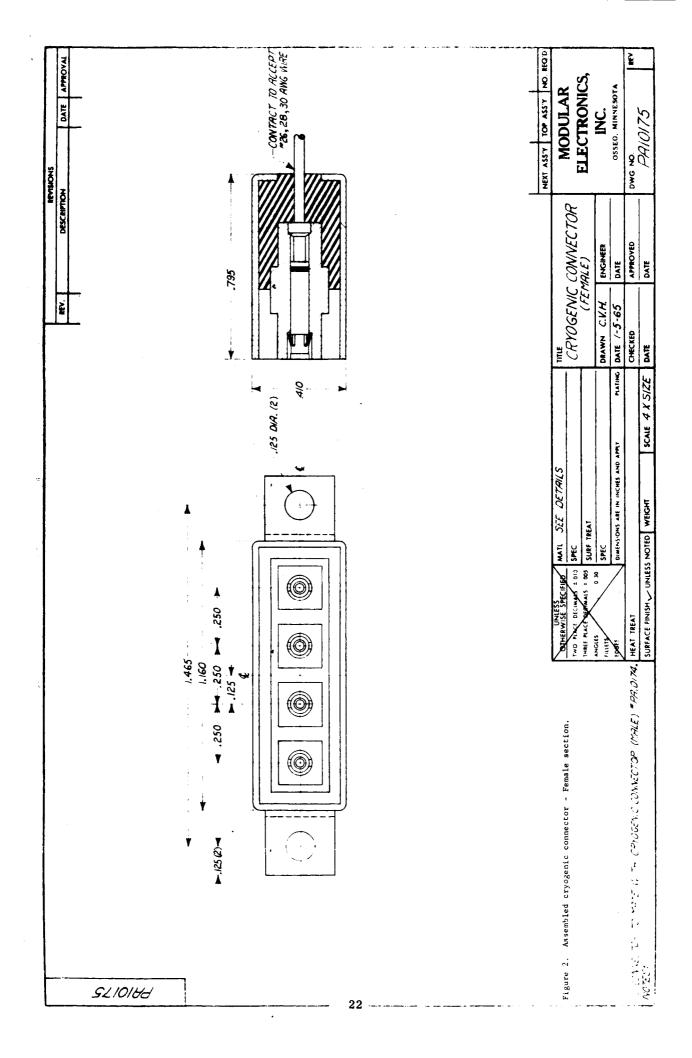
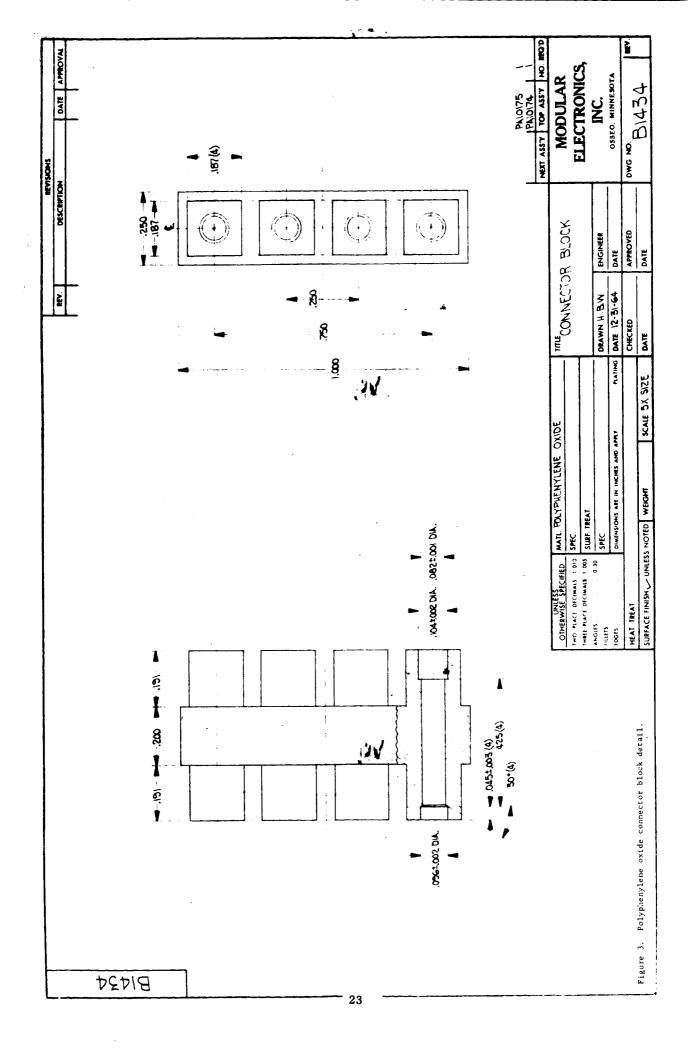
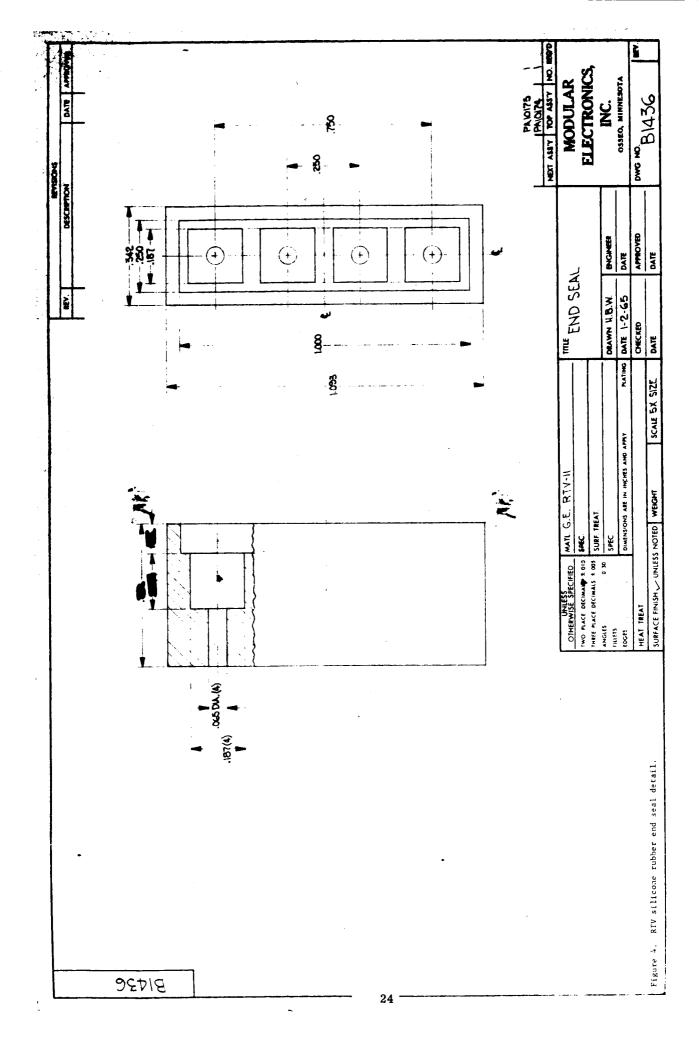


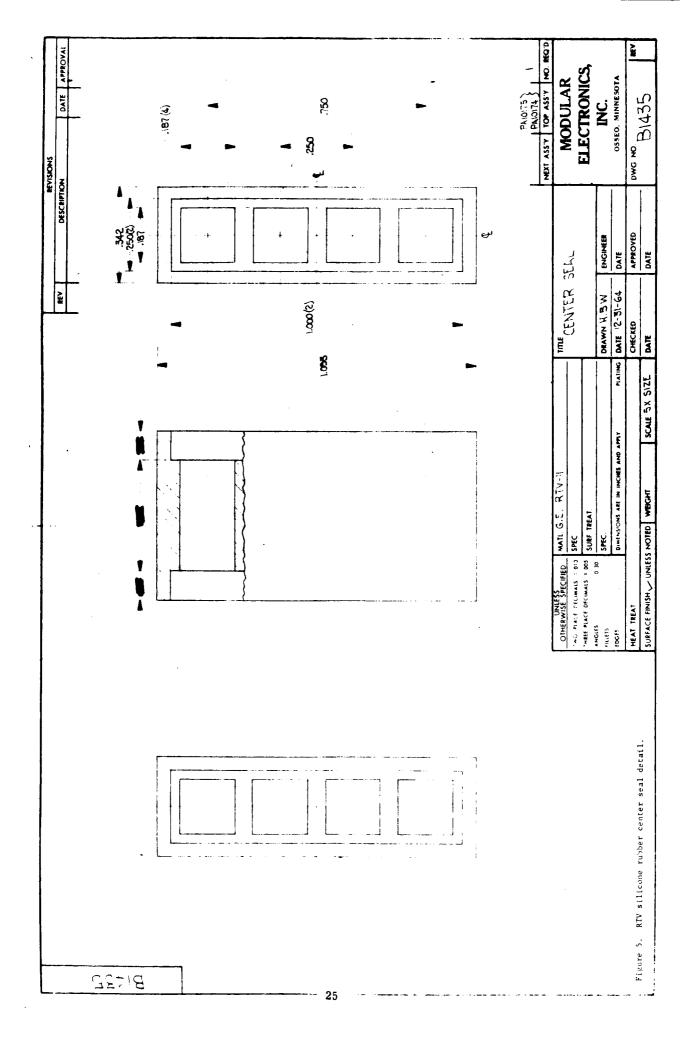
Photo. 4. Microflame torch being used to remove H-film from shielded rubber cable. -20-

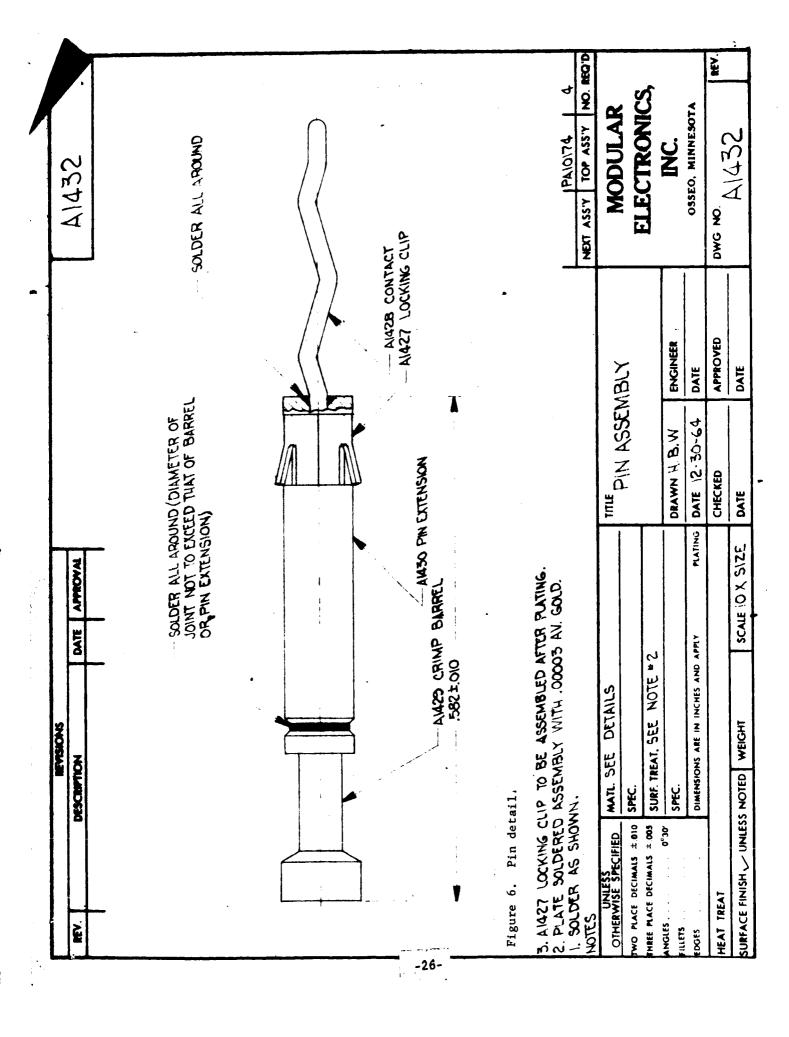












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Figure 7. Receptacle detail	letail.				
3. A1427 LOCKING CLIP TO BE ASSEMBLED AFTER PLATING. 2. PLATE SOLDERED ASSEMBLY WITH .00003 AV. GOLD.	BE ASSEMBLED AFTE BLY WITH .00003 P	JR PLATING. N. GOLD.			A . ACLAND.
1, SOLDER AS SHOWN.		•			NEXT ASS'Y TOP ASS'Y NO. REQ'D
ALESS SE SPECIFIED MATE.	SEE DETAILS		VICENSORY STORY	A ACCENSOIV	MODULAR
TWO PLACE DECIMALS # 010 SPEC.			חברבי וארו	L Macimians	
THREE PLACE DECIMALS # 005 SURF. TREAT,	EAT. SEE NOTE #2				ELECTRONICS
FILLETS			DRAWN H.B.W	ENGINEER	ن کار
EDGES DIMENSHO	DIMENSIONS ARE IN INCHES AND APPLY	PLATING	DATE 12-31-64	DATE	OSSEO, MINNESOTA
HEAT TREAT			CHECKED	APPROVED	DWG NO.
SURFACE FINISH WUNLESS NOTED	WEIGHT	SCALE 10 X SIZE	DATE	DATE	A1433

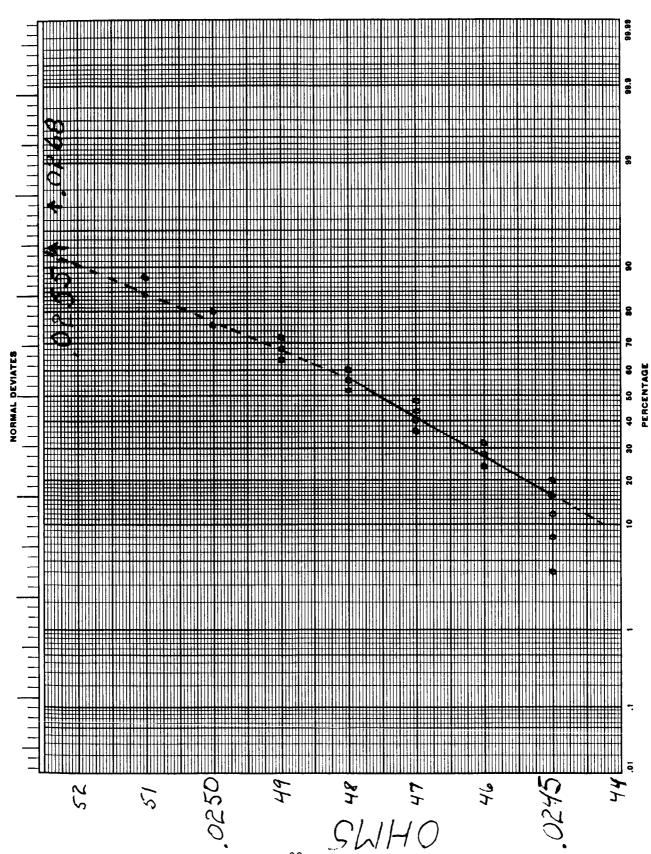


Figure 8. Resistance of test wires and connections at $23^{\circ}\mathrm{C}$.

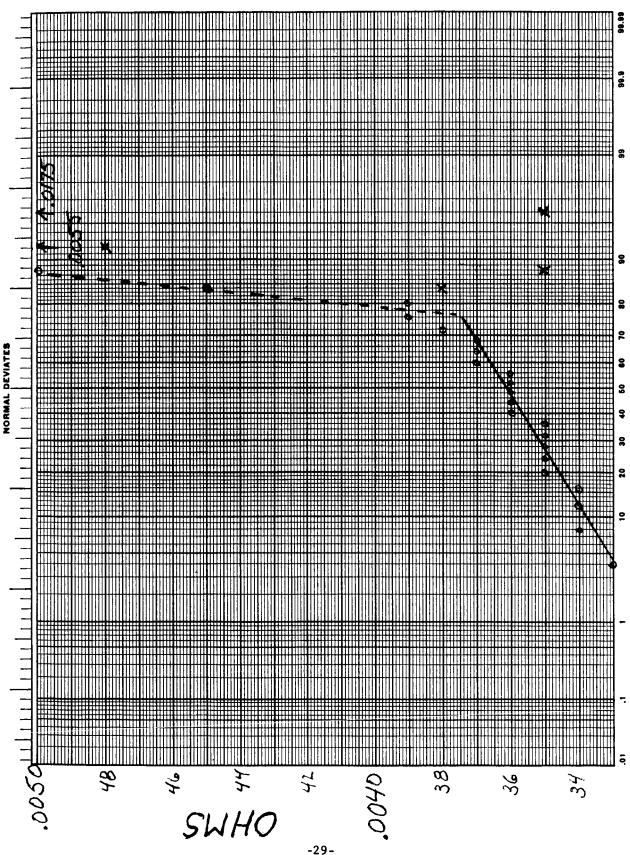


Figure 9. Resistance of test wires and connections at -196°C. X indicates value after mechanical "rework".